U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE ATTORNEY'S DOCKET NUMBER FORM-PTO-1390 (Rev. 12-29-99) TRANSMITTAL LETTER TO THE UNITED STATES 003300-852 DESIGNATED/ELECTED OFFICE (DO/EO/US) U.S. APPLICATION NO known, see 37 C.F.R 1 5) **CONCERNING A FILING UNDER 35 U.S.C. 371** unassigned INTERNATIONAL APPLICATION NO. INTERNATIONAL FILING DATE PRIORITY DATE CLAIMED 29 March 1999 PCT/SE00/00597 28 March 2000 TITLE OF INVENTION A PROCESS FOR STERILIZING A BIOLOGICALLY CONTAMINATED ENCLOSURE APPLICANT(S) FOR DO/EO/US SHOAA ABDUL RAHMAN and RAÁFAT KITTANEH Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information: This is a FIRST submission of items concerning a filing under 35 U.S.C. 371. 1. This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371. 2.  $\boxtimes$ This is an express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination 3. until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and the PCT Articles 22 and 39(1).  $\boxtimes$ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date. 4.  $\boxtimes$ 5. A copy of the International Application as filed (35 U.S.C. 371(c)(2)) is transmitted herewith (required only if not transmitted by the International Bureau).  $\boxtimes$ has been transmitted by the International Bureau. b. is not required, as the application was filed in the United States Receiving Office (RO/US) A translation of the International Application into English (35 U.S.C. 371(c)(2)). 6.  $\boxtimes$ 7. Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3)) are transmitted herewith (required only if not transmitted by the International Bureau). have been transmitted by the International Bureau. have not been made; however, the time limit for making such amendments has NOT expired. have not been made and will not be made. A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)). X 9. An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)). (Signed Declaration will follow). A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)). Items 11. to 16. below concern other document(s) or information included: 11. An Information Disclosure Statement under 37 CFR 1.97 and 1.98. An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included. 12. 13. A FIRST preliminary amendment. A SECOND or SUBSEQUENT preliminary amendment. A substitute specification.

A certified copy of Swedish Application No. 9901137-1, filed 29 March 1999, was submitted during the international phase of the

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A change of power of attorney and/or address letter.

examination. Thus, the claim for priority has been perfected.

Other items or information:

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d. The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 02-4800. A duplicate copy of this sheet is enclosed.							
NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.					137(a) or (b))		
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Benton S. Duffett, Jr.  BURNS, DOANE, SWECKER & MATHIS, L.L.P.  P.O. Box 1404					//wy	<u> Ν.</u>	
		, Virginia 22313-1404		Benton S. Duffett, . NAME	Jr.	·····	
	Filed: Sep	otember 28, 2001		22,030 REGISTRATION NUMBER	 R		

### 09/937851 410 Rec'd PCT/PTO 2 8 SEP 2001

Patent Attorney's Docket No. <u>003300-852</u>

#### IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of	)	
SHOAA ABDUL RAHMAN et al.	)	Group Art Unit: (unassigned)
Application No.: (unassigned)	)	Examiner: (unassigned)
Filed: September 28, 2001	)	
For: A PROCESS FOR STERILIZING A BIOLOGICALLY CONTAMINATED ENCLOSURE	)	
21,0200010	,	

#### **PRELIMINARY AMENDMENT**

Assistant Commissioner for Patents Washington, D.C. 20231

Sir:

This is a national phase application of International Application No.

PCT/SE00/00597, filed March 28, 2001.

Please amend the Application as indicated.

#### IN THE ABSTRACT:

Please add the Abstract of the Disclosure that is provided on a separate sheet.

#### IN THE CLAIMS:

Kindly replace Claims 1, 3, 5 and 6 as follows:

1. (Amended) A process for sterilizing a biologically contaminated enclosure in which the enclosure is brought into contact with formaldehyde, said formaldehyde being released from formalin by heat generated by an exothermic reaction occurring in the

presence of said formalin, the exothermic reaction being provided by addition of at least one reagent to said formalin in amounts sufficient to generate heat for releasing formaldehyde from said formalin, wherein said at least one reagent comprises a first reagent (A) and a second reagent (B), said reagent (A) comprising hexamethylenetetramine, and said second reagent (B) comprising a peroxide compound or a precursor thereof.

- 3. (Amended) A process of claim 1, wherein to evaporate formaldehyde gas from every 10 ml up to maximum 3500 ml of formalin there are 180 to 210 grams of hexamethylenetetramine.
- 5. (Amended) A process of claim 1, wherein the ratio between the reagents (A, B) is 0.7 to 1.5.
- 6. (Amended) A process of claim 1, wherein the formalin has a concentration of 10 to 40% formaldehyde gas.

Please add the following new Claims 7 to 20:

7. (New) A process for sterilizing a biologically contaminated enclosure in which the enclosure is brought into contact with formaldehyde, said formaldehyde being released from formalin by heat generated by an exothermic reaction occurring in the presence of said formalin, the exothermic reaction being provided by addition of at least one reagent to said formalin in amounts sufficient to generate heat for releasing

formaldehyde from said formalin, wherein said at least one reagent comprises a first reagent (A) and a second reagent (B), said reagent (A) comprising hexamethylenetetramine, in admixture with sulphur sublime, red iron oxide, silica and citric acid, and said second reagent (B) comprising a peroxide compound or a precursor thereof.

- 8. (New) A process of claim 2, wherein said reagent (B) comprises a hydrogen peroxide solution.
- 9. (New) A process of claim 2, wherein to evaporate formaldehyde gas from every 10 ml up to maximum 3500 ml of formalin 180 to 210 grams of hexamethylenetetramine.
- 10. (New) A process of claim 7, wherein to evaporate formaldehyde gas from every 10 ml up to maximum 3500 ml of formalin the following ranges of constituents of reagent (A) are utilized: up to 10 grams of sulphur sublime, up to 10 grams of red iron oxide, up to 5 grams silica, up to 5 grams of citric acid, and 180 to 210 grams of hexamethylenetetramine.
- 11. (New) A process of claim 2, wherein the ratio between the reagents (A, B) is 0.7 to 1.5.

- 12. (New) A process of claim 3, wherein the ratio between the reagents (A, B) is 0.7 to 1.5.
- 13. (New) A process of claim 4, wherein the ratio between the reagents (A, B) is 0.7 to 1.5.
- 14. (New) A process of claim 7, wherein the ratio between the reagents (A, B) is 0.7 to 1.5.
- 15. (New) A process of claim 8, wherein the ratio between the reagents (A, B) is 0.7 to 1.5.
- 16. (New) A process of claim 9, wherein the ratio between the reagents (A, B) is 0.7 to 1.5.
- 17. (New) A process of claim 2, wherein the formalin has a concentration of 10 to 40% formaldehyde gas.
- 18. (New) A process of claim 7, wherein the formalin has a concentration of 10 to 40% formaldehyde gas.

- 19. (New) A process of claim 8, wherein the formalin has a concentration of 10 to 40% formaldehyde gas.
- 20. (New) A process of claim 9, wherein the formalin has a concentration of 10 to 40% formaldehyde gas.

#### **REMARKS**

The present Amendment modifies the claim format and eliminates the use of multiple dependency.

The examination and allowance of the Application respectfully is requested.

Respectfully submitted,

BURNS, DOANE, SWECKER & MATHIS, L.L.P.

Benton S. Duffett, Jr. Registration No. 22,030

P.O. Box 1404 Alexandria, Virginia 22313-1404 (703) 836-6620

Date: September 28, 2001

# Attachment to Preliminary Amendment dated September 28, 2001 Marked-up Claims 1, 3, 5 and 6

- 1. (Amended) A process for sterilizing a biologically contaminated enclosure in which the enclosure is brought into contact with formaldehyde, said formaldehyde being released from formalin by heat generated by an exothermic reaction occurring in the presence of said formalin, the exothermic reaction being provided by addition of at least one reagent to said formalin in amounts sufficient to generate heat for releasing formaldehyde from said formalin, [characterized in that] wherein said at least one reagent comprises a first reagent (A) and a second reagent (B), said reagent (A) comprising hexamethylenetetramine, [optionally in admixture with sulphur sublime, red iron oxide, silica and citric acid,] and said second reagent (B) comprising a peroxide compound or a precursor thereof.
- 3. (Amended) A process of [any one of claims 1-2] <u>claim 1</u>, wherein to evaporate formaldehyde gas from every 10 ml up to maximum 3500 ml of formalin [the following ranges of constituents of reagent (A) are needed: (0-10) grams of sulphur sublime, (0-10) grams of red iron oxide, (0-5) grams silica, (0-5) grams of citric acid and (180-210)] there are 180 to 210 grams of hexamethylenetetramine.
- 5. (Amended) A process of [any one of claims 1-4] <u>claim 1</u>, wherein the ratio between the reagents (A, B) is [0.7-1.5] <u>0.7 to 1.5</u>.

#### Attachment to Preliminary Amendment dated September 28, 2001

#### Marked-up Claims 1, 3, 5 and 6

6. (Amended) A process of [any one of claims 1-5] <u>claim 1</u>, wherein the formalin has a concentration of [10-40] <u>10 to 40</u>% formaldehyde gas.

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#### PROCESS FOR STERILIZING A BIOLOGICALLY CONTAMINATED ENCLOSURE

The present invention relates to a process for sterilizing a biologically contaminated enclosure and particularly to a safe and easy chemical process for a wellcontrolled release or fumigation of formaldehyde gas from formalin. The process is intended for sterilizing biologically contaminated enclosures such as animal houses, hatcheries, feed stores, feed bins, feed tanks, feed mills, hospitals, medical instruments or other hard to reach areas in which the slowly and well controlled release of formaldehyde gas from easily available formalin solution is responsible for the complete sterilization by maximizing the excellent killing effects against bacteria, viruses and fungi.

#### 15 BACKGROUND OF THE INVENTION

#### Discussion of related art

It is well known that formaldehyde gas is the mostly used gas in the sterilization of livestock buildings. It is an effective disinfectant against vegetative bacteria, fungi, spores and viruses if an adequate time of exposure and not less than 70% humidity are provided (Remington page 1171, Cooper & Mason 1964).

It can be easily absorbed by surfaces in both its 25 gaseous or liquid state, as it has strong penetration power which increases in completely closed spaces. In a study (Sykes, 1972) it was proved that formaldehyde can attain complete sterilization to a well closed room within two hours at 70% relative humidity.

Its mode of action on living cells is by reaction with the cellular protein and (DNA, RNA) amino acids (Russel, 1976).

Formalin, as a commercially available product, is an aqueous solution containing up to 37% by weight of formaldehyde, HCHO (30.03), with methanol added to prevent

polymerization. The solution is extensively used for disinfecting rooms, which have been subjected to infection, by:

- 5 1. Reaction of formalin with half its weight of potassium permanganate.
  - 2. Spraying it on sheets hung in the room.
- 10 3. Releasing formaldehyde vapor from formalin into the room by a heat generator or heater.
  - 4. Subliming paraformaldehyde powder at 218°C using a heater.

There are disclosed in WO 97/23247 A1 antiperspirant pads which are impregnated with hexamethylenetetramine in an acidic or neutral medium for releasing formaldehyde.

From STN International, File Caplus, Accession no. 1985:492906, Document no. 103:92906, RO 85306 B, it is known to disinfect technological spaces by using formal-dehyde which is rapidly released from a formalin solution by a strong exothermic reaction due to the addition of KMnO<sub>4</sub>.

In US patent no. 4356179 formaldehyde products are used as agricultural fungicides. The products can for example be obtained by adding hexamethylenetetramine to formaldehyde.

All the above mentioned methods have many disadvantages. Using potassium permanganate to evaporate formaldehyde results in a very vigorous and dangerous reaction,
which is completed within few seconds, and this does not
give enough time for the worker to escape safely and many
fatal accidents have been reported in real life.

It can be proved that spraying formalin on sheets is non-reproducible and does not release enough formaldehyde gas as it is temperature-dependent as can be seen from the following table

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Concentration and type of formaldehyde	Time néeded to kill spores	Time needed to kill bacteria
Formaldehyde gas	2 hours	2 hours
8% formalin	18 hours	18 hours
0.5% formalin	2-4 days	6-12 hours

Formaldehyde gas is thus described as being a sterilizing agent, while formalin (10% strength) is described as being an antiseptic agent; hence there is a need to use the formaldehyde gas as a sterilizer rather than using the formalin, as formalin is time consuming and requires diligent application through decontamination.

Reference: Remington's Pharmaceutical Sciences, 18th. Ed., pages 1171, 1470. Merck Index 11th Ed.

Further, contaminated, highly sophisticated electronic and dental equipment may be damaged by sterilizing liquid agents like those described in US Patent 31779 or by heat or autoclaving etc. US Patent 5552112 introduced a new method for sterilizing a metallic surgical instrument with microwave radiation but the method suffers from non-availability of microwave source to everyone and high cost. As mentioned in US Patent 5552112, gas sterilization with an ethylene oxide mixture is acceptable for both hanpieces and burs. However, this is impractical because of cost of equipment, long sterilization and aeration times involved, and cost of providing adequate protection for personnel. Alkaline glutaraldehyde (2%) as mentioned by Boucher in US Patent 3912450, is used to sterilize equipment, but it must be used for 10 hours to kill spore-forming organisms or tuberculosis microorganisms and is irritating to tissue.

Many workers could successfully release formaldehyde gas using generators or heaters and some patents have been published like US Patents: 4585624, 665794, 1837264, 2993832, 3694146, 3816074, 3898038 and 4166087. All these patents describe different apparatus systems for vaporiz-

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ing formaldehyde and dispensing it into the enclosure to be sterilized for contact with contaminants. However, such methods suffer from high cost of instruments, maintenance, availability, complexity and restricted volume of formalin to be evaporated. No work has ever been known to use a safe chemical process for the evaporation of formaldehyde as an alternative for the unsafe usage of potassium permanganate or other conventional methods.

#### 10 SUMMARY OF THE INVENTION

The present invention provides an ideal, chemical and safe process for releasing formaldehyde gas from a formalin solution by evaporation using heat generated by an exothermic reaction in order to simplify and optimize the use of formaldehyde gas in sterilizing closed spaces, like houses, buildings for housing animals, hospitals, operating rooms, stores, hotels, bath rooms or any object needing to be sterilized.

It is therefore an object of the present invention to provide a process for sterilizing enclosures, such as animal houses, hatcheries, feed stores, feed bins, feed tanks, feed hauling truck bins or tanks, feed mills or other hard to reach areas, or rooms in hospitals, clinics, research laboratories and the like by chemically releasing gaseous formaldehyde into the enclosure for contact with contaminants.

The present invention is based on the finding that the release of formaldehyde gas can be easily provided by heat generated by an exothermic reaction occurring in the presence of formalin. The released formaldehyde gas can be used for sterilizing closed spaces of the kind exemplified above. Exothermic chemical reactions involving various reagents are known to one skilled in the chemical art. In general, a chemist can chose any reagents for such exothermic reactions. Needless to say, it is advantageous to chose reagents, which are easily available and cheep. Once chosen the amounts of reagents sufficient to

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generate heat for releasing gaseous formaldehyde for sterilization from formalin are easily determined by one skilled in the art. As an example of appropriate reagents there can be mentioned hexamethylenetetramine and peroxide compounds or precursors of peroxide compounds, which when mixed, and in the presence of formalin, bring about an exothermic reaction releasing formaldehyde gas for sterilizing.

According to the invention the exothermic reaction is provided by addition of reagents to the formalin in amounts sufficient to generate heat for releasing formal-dehyde from the formalin. In accordance therewith, the reagents comprise a first reagent A and a second reagent B, said reagent A comprising hexamethylenetetramine, optionally in admixture with sulphur sublime, red iron oxide, silica, preferably that sold under the tradename Aerosil, and citric acid, and the second reagent B comprising a peroxide compound, such as hydrogen peroxide, or a precursor thereof.

According to a most preferred embodiment, the reagent A comprises Methenamine (hexamethylenetetramine) and the reagent B comprises a hydrogen peroxide solution.

Mostly preferred, said solution contains 10-50% hydrogen peroxide.

In accordance with the present invention the formallin, for example having a concentration of 10-40% formaldehyde gas, is mixed with the reagent A comprising hexamethylenetetramine and the reagent B, for example hydrogen peroxide. As explained below, the temperature of the
reaction solution will increase spontaneously by the exothermic chemical reaction and the production of formaldehyde gas starts effectively at 60°C. The temperature
rises and release of formaldehyde gas reaches the maximum
value at 90°C.

As non-limiting examples of peroxide compounds the following can be mentioned: ammonium peroxosulfate, po-tassium peroxodisulfate, hydrogen peroxide, acetyl perox-

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ide, benzoyl peroxide and cumene hydroperoxide.

According to a most preferred embodiment, wherein said reagents comprise hexamethylenetetramine and hydrogen peroxide the ratio between these is within the range of 0.7-1.5.

#### DETAILED DESCRIPTION OF THE INVENTION:

The present invention is illustrated by the following example:

To evaporate 10 ml up to maximum 3500 ml (preferably 2000 ml) of formalin containing 10%-40% of formaldehyde gas in water:

The above formalin quantity is mixed with 100g-400g, preferably 200 g powder (A) which consists of five ingredients as follows:

Ingredient	Range in grams	Preferably in grams
Sulphur sublime	0-10	0.30
Iron oxide, red	0-10	0.30
"Aerosil"	. 0-5	0.40
Citric acid	0-5	4.00
Methenamine	180-210	195.00

In case of sterilizing highly sophisticated medical or dental instruments or other similar objects Methenamine (hexamethylenetetramine) without the rest of chemicals shown in the above table should be used.

After mixing the above quantity of powder (A) with the above amount of formalin, (100ml-400ml, best results with 200ml) liquid (B) is added.

It is the powder part, which controls the reaction, and provides enough time for the operator before the onset of the evaporation process.

Liquid (B) is hydrogen peroxide having a concentration of 10%-50% (best results with 50%). It is the heat generated by mixing powder (A) and liquid (B) which helps

in heating and evaporation or controlled fumigation of formalin.

After about five minutes, the temperature of the solution increases spontaneously by the exothermic chemical reaction and the production of formaldehyde gas starts effectively at 60° C. With the rise of the temperature of the exothermic reaction the evaporation of formaldehyde gas becomes very strong and reaches the maximum at 90° C (after about 10 minutes).

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#### EXAMPLES:

#### Example 1:

In order to illustrate the invention the following non-limiting example is given:

A 4000 m³ (10000 bird capacity) chicken house was chosen to be sterilized immediately after the complete evacuation of the building and before the new bird cycle. The house was left as such without further cleaning. Swabs were taken from different representative areas of the house (ceiling, flour etc.). All windows and openings were tightly closed. The house was humidified with water to obtain at least 70% relative humidity. The chosen formalin amount for the experiment was 2 lit per 100m³, so 40 lit of (37%) formaldehyde solution were divided into 20 plastic containers (10 lit capacity each) such that each container contains 2 lit of formalin. The containers were placed evenly in the house. To each container 200 g of powder (A) were added and consisting of:

	<u>Ingredient</u>	Quantity (g)
30	Sulfur Sublime	0.30
	Iron oxide, red	0.30
	"Aerosil"	0.40
	Citric acid	4.00
	Methenamine	195.00
35	TOTAL	200.00

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Powder (A) was mixed thoroughly with formalin and thereafter 200 ml of liquid (B) (50% hydrogen peroxide) were added to every container and mixed.

In every container solution temperatures were recorded against time, and the following average solution

temperatures were obtained ag-	allist time.
Time in minutes	Solution Temperature °C
·	
3	40
5	. 60
7	80
10	95
30	· 95
40	85
50	75
60	65
100	40

Formaldehyde started evaporating effectively when the temperature of the solution reached 60° C after 5 minutes of mixing all ingredients together.

After 10 minutes the evaporation became extensive (boiling).

This temperature was attained for 20 minutes before it declined and reached room temperature after two hours.

The chicken house was kept closed and empty for 48 hours after which it was well ventilated and swabs were taken again randomly and analyzed for total fungal and bacterial count.

	Average count after sterili-		
Average count before	Average count after scentill		
sterilization	zation		
D 0 0 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2			
1000,000 colonies/g	2 colonies/g		

#### 20 Example 2:

Calculate the size of the house and then put in separate containers (20 liters capacity) two liters of Formalin for each 100 cubic metre. Add 200 g of reagent A by mixing with the standard Formalin in all containers; steer the mix after adding the powder until it is com-

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pletely dissolved, then add 200 ml of Reagent B to the mixture and leave the house. After five minutes evaporation starts and the release of formaldehyde gas continue for more than four hours and it is preferable to keep the house closed overnight. The production of formaldehyde gas starts effectively at 60°C. The evaporation becomes very strong and reaches the maximum at 90°C after 10 minutes. Fumigation works best at temperatures above 18°C, therefore the house temperature should be maintained above this level and the humidity should be around 70%. The composition of reagent A is sulphur, iron oxide, dicalcium phosphate and hexamethylenetetramine. Reagent A controls the formaline evaporation reaction and provides a suitable time before the onset of evaporation. Reagent B is a liquid comprising 50% hydrogen peroxide which is sufficient to evaporate two litres of 37% formalin solution. Reagent B assists in heating and functions as a catalyst for evaporation of formalin.

Reagent A is to be kept in a cool place at a temperature below 30°C, out of reach of children and feed stock places. Reagent B is also to be kept in a cool place at a temperature below 30°C, out of reach of children and feed stock places.

#### Example 3:

The procedure of this example was carried out in the same manner as in example 2 with the exception of that the composition of reagent A is 100% hexamethylenetetramine powder. The results of this experiment were also excellent as in the aforementioned experiments.

The present invention, therefore, is well suited and adapted to attain the intended objects and has the advantages and features mentioned as well as others inherent therein. The foregoing description is provided to illustrate the invention, and is not to be construed as a limitation.

#### REFERENCES

- Russell, A.D. 1976. Inactivation of non-sporing bacteria by gases. SOC.APPL.Bacterial., 5, 61-68.
  - 2. Cooper, E.A., and Mason, J. 1964. Studies of selective bactericidal action. J.HYG, 26, 118-126.

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- 3. Sykes, M.K. 1972 . Sterilization of ventilators. Int. Anesthesiol. Clin., 10, 131.
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#### CLAIMS

- 1. A process for sterilizing a biologically contaminated enclosure in which the enclosure is brought into contact with formaldehyde, said formaldehyde being released from formalin by heat generated by an exothermic reaction occurring in the presence of said formalin, the exothermic reaction being provided by addition of at least one reagent to said formalin in amounts sufficient 10 to generate heat for releasing formaldehyde from said formalin, characterized in that said at least one reagent comprises a first reagent (A) and a second reagent (B), said reagent (A) comprising hexamethylenetetramine, optionally in admixture with sulphur sub-15 lime, red iron oxide, silica and citric acid, and said second reagent (B) comprising a peroxide compound or a precursor thereof.
  - 2. A process of claim 1, wherein said reagent (B) comprises a hydrogen peroxide solution.
  - 3. A process of any one of claims 1-2, wherein to evaporate formaldehyde gas from every 10 ml up to maximum 3500 ml of formalin the following ranges of constituents of reagent (A) are needed: (0-10) grams of sulphur sublime, (0-10) grams of red iron oxide, (0-5) grams of silica, (0-5) grams of citric acid and (180-210) grams of hexamethylenetetramine.
  - 4. A process of claim 2, wherein the solution contains 10-50% hydrogen peroxide.
  - 5. A process of any one of claims 1-4, wherein the ratio between the reagents (A, B) is 0.7-1.5.
    - 6. A process of any one of claims 1-5, wherein the formalin has a concentration of 10-40% formaldehyde gas.

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## COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY (Includes Reference to Provisional and PCT International Applications)

Attorney's Docket No. 003300-852

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I acknowled 37, Code of I hereby cla or inventor	dge the duty to di f Federal Regulat aim foreign priori 's certificate or o sted below and ha	referred to above. sclose to the Office all information ions, §1.56.  ty benefits under Title 35, United 5 any PCT international application we also identified below any foreig esignating at least one country other date before that of the application	States Code, §119 (a)-(e) of any f (s) designating at least one countr n application(s) for patent or inve er than the United States of Ameri	oreign application(s) for patent y other than the United States o ntor's certificate or any PCT		
internations			TV 01 A1340 LINDED 05 11 C 0			
internationa subject mat	FIGN/PCT APP	ICATION(S) AND ANY PRIORI	I Y CLAIMS UNDER 35 0.5.	C. §119:		
internationa subject mat RIOR FOR	EIGN/PCT APPI		DATE OF FILING	PRIORITY CLAIMED		
internationa subject mat RIOR FOR COU (if PCT, inc	UNTRY dicate "PCT")	APPLICATION NUMBER	DATE OF FILING (day, month, year)	PRIORITY CLAIMED UNDER 35 U.S.C. §119		
internationa subject mat RIOR FOR	UNTRY dicate "PCT")		DATE OF FILING	PRIORITY CLAIMED UNDER 35 U.S.C. §119  X Yes _No		
internationa subject mat RIOR FOR COU (if PCT, inc	UNTRY dicate "PCT")	APPLICATION NUMBER	DATE OF FILING (day, month, year)	PRIORITY CLAIMED UNDER 35 U.S.C. §119  _X Yes _No _Yes _No		
internationa subject mat RIOR FOR COU (if PCT, inc	UNTRY dicate "PCT")	APPLICATION NUMBER	DATE OF FILING (day, month, year)	PRIORITY CLAIMED UNDER 35 U.S.C. §119  X Yes _No _Yes _No _Yes _No _Yes _No		
internations subject mat subject mat RIOR FOR COL (if PCT, inc. Swede	UNTRY dicate "PCT") N	APPLICATION NUMBER 9901137-1	DATE OF FILING (day, month, year) 29 March 1999	PRIORITY CLAIMED UNDER 35 U.S.C. §119 X_YesNoYesNoYesNoYesNoYesNo		
international subject mat subject mat RIOR FOR COL (if PCT, inc. Swede	UNTRY dicate "PCT") N	APPLICATION NUMBER	DATE OF FILING (day, month, year) 29 March 1999	PRIORITY CLAIMED UNDER 35 U.S.C. §119  _X_YesNoYesNoYesNoYesNoYesNo		

#### COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY (CONTINUED) (Includes Reference to Provisional and PCT International Applications)

Attorney's Docket No. 003300-852

I hereby claim the benefit under Title 35, United States Code, §120 of any United States applications(s) or PCT international application(s) designating the United States of America that is/are listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in that/those prior application(s) in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose to the Office all information known to me to be material to the patentability as defined in Title 37, Code of Federal Regulations §1.56, which became available between the filing date of the prior application(s) and the national or PCT international filing date of this application:

PRIOR U.S. APPLICATIONS OR PCT INTERNATIONAL APPLICATIONS DESIGNATING THE U.S. FOR BENEFIT UNDER 35 U.S.C. 120:

U.S. APPLICATIONS			STATUS (check one)		
U.S. APPLICATION NUMBER		U.S. FILING DATE	PATENTED	PENDING	ABANDONED
PCT	APPLICATIONS DESIGNAT	TING THE U.S.			
PCT APPLICATION NO.	PCT FILING DATE	U.S. APPLICATION NUMBERS			
SE00/00597	28 March 2000				
					<u> </u>

I hereby appoint the following attorneys and agent(s) to prosecute said application and to transact all business in the Patent and Trademark Office connected therewith and to file, prosecute and to transact all business in connection with international applications directed to said invention:

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Robert S. Swecker	19
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Benton S. Duffett, Jr.	22
Norman H. Stepno	22
Ronald L. Grudziecki	24
Frederick G. Michaud, Jr.	26
Alan E. Kopecki	25
Regis E. Slutter	26
Samuel C. Miller, III	27
Robert G. Mukai	28
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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

2. [	ONBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY (CONTINUED) actudes Reference to Provisional and PCT International Applications)		ATTORNEY'S DOCKET NO. 003300-852	
·	FULL NAME OF SOLE OR FIRST INVENTOR	SIGNATURE Q		DATE 20 Nov 20
	Shoaa Abdul <u>RAHMAN</u>	1 FOR MORM	CITIZENSHIP	
	Lund, Sweden SEX	2.	· Jordan	
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0	FULL NAME OF SECOND JOINT INVENTOR, IF ANY Raáfat KIITANEH	SIGNATURE Lac	CITIZENSHIP	DATE 20 Nov 20
	RESIDENCE AMMAN, JORDAN JORDAN JORDAN		Jordan	
	Wadi sagra StKalbona Building, 6th Floor, Flat No. 7, AMMAN, JORDAN			
	FULL NAME OF THIRD JOINT INVENTOR, IF ANY	SIGNATURE		DATE
	RESIDENCE		CITIZENSHIE	
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	FULL NAME OF FOURTH JOINT INVENTOR, IF ANY	SIGNATURE		DATE
	RESIDENCE		CITIZENSHII	
	POST OFFICE ADDRESS			
	FULL NAME OF FIFTH JOINT INVENTOR, IF ANY	SIGNATURE		DATE
	RESIDENCE		CITIZENSHI	P
	POST OFFICE ADDRESS			
	FULL NAME OF SIXTH JOINT INVENTOR, IF ANY	SIGNATURE		DATE
	RESIDENCE		CITIZENSHI	P
	POST OFFICE ADDRESS			T = 100
	FULL NAME OF SEVENTH JOINT INVENTOR, IF ANY	SIGNATURE		DATE
	RESIDENCE		CITIZENSH	LP
	POST OFFICE ADDRESS			DATE
	FULL NAME OF EIGHTH JOINT INVENTOR, IF ANY	SIGNATURE	- CONTRACTOR	
	RESIDENCE		CITIZENSH	<u> </u>
	POST OFFICE ADDRESS			Dir
	FULL NAME OF NINTH JOINT INVENTOR, IF ANY	SIGNATURE		DATE
	RESIDENCE		CITIZENSH	Щ² 
	POST OFFICE ADDRESS			